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An Investigation of the Stability of and Insulation Leakage in Some High Temperature Resistance Thermometers: An Interim Report

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

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An Investigation of the Stability of and Insulation Leakage in Some High Temperature Resistance Thermometers: An Interim Report

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FOREWORD

In the interest of informing the public on program developments and technical advances within the Division, we are increasing our publications activities, beyond the customary formal publication of complete pieces of work, through the media of NBS Technical Notes and NBS Reports. The former provide the means to deal with the subject matter at greater depth than normally tolerated by today's journal editors and the latter afford an opportunity to describe informally the progress toward current goals where the work is incomplete.

This is a progress report. The work is incomplete and is continuing. Results and conclusions are not necessarily those that will be included in the final, formal publications. The period covered herein is the calendar year 1972.

H. H. Plumb, Chief
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Data are presented concerning the stability of high temperature resistance thermometers. Two types of platinum and one type of tungsten thermometer were tested at 1065 °C and 960 °C. Some of the platinum thermometers were also tested at 900 °C and at the freezing point of zinc. Results of tests are also given on the insulation resistance of sensor supports and lead assemblies for the two types of platinum thermometers. A new design for a zinc-point cell is discussed briefly. Suggestions for future work are indicated.

Key words: Freezing point of zinc; high temperature resistance thermometer; insulation resistance; platinum resistance thermometer; resistance thermometer; stability; tungsten thermometer; zinc.

1. Introduction

Two main paths of investigation have been followed recently in the high temperature platinum resistance thermometry laboratory at the National Bureau of Standards; one is thermometer stability and the other is thermometer insulation resistance. Drifts and instability in thermometer resistance have long been problems for high temperature resistance thermometers when they are cycled between high temperatures (up to 1100 °C) and room temperature. Typically, a thermometer is subjected to a high temperature for a short time (e.g., one hour) and then quenched fairly rapidly upon removal from the furnace or the freezing point apparatus. It would be ideal if the resistance of a thermometer were independent of the thermal treatment it received, but strain and quenching are two well known mechanisms which can change the thermometer resistance. Researchers have tried to eliminate strain through design. Recent changes in annealing procedures [1] have eliminated much of the instability due to quenching found in thermometers, but drift and unstable behavior from these and other sources remain problems. In an effort to test the stability of various configurations and materials, we have conducted some long-range stability tests on three styles of thermometers.

2. High Temperature Stability

2.1. Thermometer Designs

We had available six thermometers of the birdcage design [2]. The elements of these thermometers contained synthetic sapphire insulators and were designated <u>h</u>igh <u>t</u>emperature <u>s</u>ynthetic <u>s</u>apphire or HTSS. The lead wires were strung through small silica glass or alumina tubes and were separated by silica glass, synthetic sapphire, or alumina disks. The insulating disks were more closely spaced near the sensing element of the thermometer to reduce radiation losses. Three of these thermometers (HTSS-14, HTSS-15, and HTSS-16) were relatively old (1960), but had not been used for several years. (HTSS-14 was studied in [2].) The elements and leads were remounted in 800 mm long, high-purity silica-glass sheaths. This required extending the leads but the elements were not modified. Two of the thermometers (HTSS-21 and HTSS-22) were new; their elements had been made earlier but the leads had not been attached. The sixth thermometer (HTSS-19) was an old thermometer (1961) which had been used during the intercomparison of high temperature thermometers and thermocouples [3]. Table 1 contains a summary of the characteristics of the thermometers that were investigated.

Table 1. Thermometer Characteristics

Serial Number	Coil Support	Lead Assembly Insulators	R_0,Ω	Style
HTSS-14	synthetic sapphire	alumina	0.26295	Birdcage
HTSS-15	synthetic sapphire	alumina	0.26983	Birdcage
HTSS-16	synthetic sapphire	alumina	0.26099	Birdcage
HTSS-19	synthetic sapphire	alumina tubes synthetic sapphire disks	0.26048	Birdcage
HTSS-21	synthetic sapphire	silica glass	0.25778	Birdcage
HTSS-22	synthetic sapphire	alumina	0.25950	Birdcage
HTFQ-23	silica glass	silica glass	0.22921	Cross
HTFQ-24	silica glass	silica glass	0.22248	Cross
HTFQ-25	silica glass	silica glass	0.21790	Cross
W-1	tungsten	silica glass	0.2580	Steeple

Three resistance thermometers of a new element design were constructed and tested for their stability. Platinum wire, 0.4 mm diam, was wound in a bifilar helix around a cross shaped silica-glass support (fig. 1) made at the National Bureau of Standards. Thermometers of a similar design have been investigated by Sawada and Mochizuki [4]. The resistance of the thermometers at the triple point of water is nominally 0.25 Ω; these cross thermometers were designated high temperature fused quartz or HTFQ. Before assembly all silica glass parts were cleaned by an ultrasonic technique in sequential baths of trichloroethylene, methyl alcohol, and distilled water, and then fired in oxygen at 1100 °C for one hour. The sheath was cleaned by washing with a detergent and then rinsing extensively with distilled water. It was dried by heating to a low temperature. Finally, it was fired for one hour at 1100 °C in oxygen. After assembly but before being inserted in the sheath, the thermometer was soaked in aqua regia for three hours, rinsed with distilled water, and baked at temperatures up to 600 °C. After being placed in the silica-glass sheath, the thermometer was evacuated, fired in oxygen for five minutes at 1100 °C, and reevacuated. The sheath was then sealed after being filled with dry air to a pressure of one atmosphere at 1065 °C. The same general technique was followed in the construction of the birdcage thermometers.

The resistance element of the third type of thermometer was fabricated from thoria-doped tungsten wire. The element was made by R. L. Anderson while he was at the National Bureau of Standards and it was built in the style of a "steeple" thermometer [5]. The thermometer leads are W-3%Re alloy wires of 0.37 mm diam and the lead assembly insulators are silica glass disks and tubes. Initially, the element was annealed at 2000 °C for one hour before the thermometer was assembled and afterwards the fabricated thermometer, designated W-1, was vacuum baked at 800 °C and filled with argon gas. Only one tungsten resistance thermometer was tested.

2.2. Experimental Procedure

The procedure followed in testing the long-term stability was the same for the three types of thermometers. Resistance readings were first taken at the triple point of water. The thermometers were then placed in a pre-heated furnace where they remained for a specified period of time after which they were annealed. Then triple-point resistance readings were taken again. These tests differ from normal use of the thermometers in two important respects. First, the thermometers are exposed to high temperatures for a longer time than in normal usage. Second, the thermometers are not quenched before annealing.

Repeated measurements at the triple point of water without intervening heat treatment show that the average difference in 20 successive pairs of readings is 0.04 mK and the standard deviation is 0.28 mK. This represents the reproducibility of our measuring system and thermometers. One cross and four birdcage thermometers were used to obtain these data.

2.3. Results of Stability Tests

a. Results of Heat Treatment in the Region of 1065 °C

Figures 2, 3 and 4 summarize the results of tests for thermometer W-1 at 1065 °C, and for five birdcage thermometers at 1100 °C, at 1065 °C, and at the freezing point of gold. Thermometer HTSS-16 (fig. 3) was tested much longer than the others because of its unstable behavior. Thermometer HTSS-21 was resheathed twice in the course of the experiment and data is given for the most recent sheath only.

There was a relatively large initial shift in the ice-point resistance for all five birdcage thermometers as a result of the heat treatment. This is probably caused by the annealing of cold working done during construction. Thermometer HTSS-14 continued to drift rapidly for a fairly long period of time but it had been dropped while out of its sheath prior to these tests and had thereby suffered a large amount of cold working and possibly slight contamination. Thermometer HTSS-22 was the only one that exhibited an initial increase in the ice-point resistance. Thermometer HTSS-21, after the initial decrease in the ice-point resistance, continued to increase in resistance with additional exposure to high temperatures.

Figure 4 is essentially an enlargement of a part of figure 2. From it, we can see the effects of various annealing patterns. The first four points represent the resistance at the ice point following an anneal of rapid cooling from 1065 °C to 650 °C (1.5 h), one hour at 650 °C, and slow cooling to room temperature. The thermometer was not quenched from 1065 °C prior to annealing. The next six points were taken after a quench from the gold point to room temperature, one hour at 650 °C, and slow cooling to room temperature over night. The last eight points were taken after a new annealing pattern which is based on the work of Berry [1]. The thermometer was placed in a furnace at 1065 °C (following a quench from the gold point to room temperature) and the furnace temperature was slowly lowered to 450 °C at a rate of 90 °C/h. For some of these last points, the thermometer was not quenched but had been in the annealing furnace at 1065 °C for several hours when the anneal began. The shift in the ice-point resistance after the first gold point was not removed by an anneal of one hour at 650 °C followed by slow cooling. The new annealing procedure was removed most of the quenched-in shift and brought the ice-point resistance closer to its original curve.

Figure 5a gives the results of the heat treatment of the three cross thermometers at 1065 °C. These thermometers were found to be generally more stable than the birdcage thermometers and to behave remarkably similarly. They were constructed at nearly the same time from the same lots of materials and HTFQ-24 and HTFQ-25 had the same thermal history. Thermometer HTFQ-23 had been used in the insulation resistance tests prior to the long-term stability tests and was subsequently repaired and resheathed. However, this treatment does not seem to have affected it to any noticeable extent.

Figures 6a and 2 give the results of testing the tungsten thermometer, W-1, at 1065 °C. We can see from figure 6a that the thermometer had not been well stabilized when the tests began. From figure 7, we see that after approximately 400 hours at 1065 °C and above, the drift rate reached 1 μΩ/h. The rate of change of the ice-point resistance seems to be an exponential function of the time at elevated temperatures. In figure 2, it is seen that the drift rate of W-1 is approaching that of some of the birdcage thermometers.

b. Results of Heat Treatment at 960 °C and 900 °C

The results of heat treatment at 960 °C for the birdcage thermometers are given in figure 8. These data were taken after the work at 1065 °C. As might be expected, the stability is better than the stability at 1065 °C. Thermometer HTSS-16 is still relatively unstable and HTSS-14 is still drifting. This may possibly be due to its gross cold working during construction but is more likely an "equilibrium" drift rate.

From figure 5b we see that the cross thermometers are well behaved at 960 °C. Two measurements at the zinc point were taken with each thermometer between the work at 1065 °C and these data. Thermometer HTFQ-24 was found to be more stable than the others but the

range of drift rates is only 0.02 mK/h. Only HTSS-16 and HTSS-21 had such small drift rates at 960 °C and they showed greater variability.

Thermometer W-1 (fig. 6b) was found to drift considerably less as a result of heating at 960 °C than from heating at 1065 °C. These measurements followed the work at 1065 °C. Its drift rate is still the largest of all the thermometers and it is difficult to tell if the drift rate is decreasing exponentially as was the case at 1065 °C.

The ice-point resistances of the birdcage thermometers resulting from heating at 900 °C are shown in figure 8. These experiments were preceded by the work at 960 °C and by measurements at the zinc point for some of the thermometers. These six thermometers, when heated at this temperature, exhibited drift rates that were equivalent to the drift rates of the cross thermometers at 960 °C and were less variable than the cross thermometer drift rates at 960 °C.

It should be noted that the heat treatments given the birdcage and cross thermometers differed in two respects. The birdcage thermometers were never continuously at a high temperature for more than twenty hours while the cross thermometers were often at a high temperature for one hundred hours at one time. Any change in the ice-point resistance which was not time dependent would be expected to have a larger effect on the drift rate of the birdcage thermometers because they were cycled more frequently than the cross thermometers. The second difference is that the cross thermometers were never given any short cycles in the gold-point cell followed by quenching. Any quenching effects that were not removed during annealing would affect only the birdcage thermometers. However, we feel that our current annealing procedure is removing noticeable quenched-in defects.

Drift rates given in this paper are the change in the ice-point resistance (calculated from the triple point resistance and corrected to zero current) per hour of exposure at elevated temperatures. The drift rates that were obtained are summarized in table 2 for the three types of thermometers at the temperatures at which they were tested. These drift rates were computed by fitting a straight line, using the least squares procedure in OMNITAB [6], to the observed values of the ice point resistances as a function of the time of exposure to high temperatures. Also, given in table 2 are the residual standard deviations and their associated degrees of freedom. The residual standard deviation is a measure of the scatter of the data points about the straight line fitted through them; it is not a measure of the variability of the y-intercept or of the slope of the line. These variabilities are given by the standard deviations of the coefficients found for the straight line. The standard deviation of the slope (drift rate) was in all cases 30% or less of the drift rate, and in all but three cases it was 15% or less.

Table 2. Drift Rates

Serial Number	HTSS 14	HTSS 15	HTSS 16	HTSS 19	HTSS 21	HTSS 22	HTFQ 23	HTFQ 24	HTFQ 25	W-1
1065 °C										
Drift rate μΩ	−0.16	−0.03	0.21		0.11	−0.13	0.07	0.06	0.04	0.30
Drift rate mK	−0.15	−0.03	0.20		0.11	−0.12	0.08	0.07	0.05	
Residual standard deviation, mK	2.35	1.05	6.16		3.11	2.77	2.73	2.85	3.11	5.82
Degrees of freedom	18	21	39		15	21	6	6	6	8
960 °C										
Drift rate μΩ	−0.04	−0.03	−0.02	0.07	0	0.04	−0.02	0	−0.01	0.10
Drift rate mK	−0.04	−0.03	−0.02	0.07	0	0.04	−0.02	0	−0.01	
Residual standard deviation, mK	0.64	0.37	1.07	0.84	0.68	1.35	0.24	0.15	0.15	2.61
Degrees of freedom	10	10	10	10	10	10	6	6	6	4

(continued)

Table 2 (continued)

Serial Number	HTSS 14	HTSS 15	HTSS 16	HTSS 19	HTSS 21	HTSS 22	HTFQ 23	HTFQ 24	HTFQ 25	W-1
900 °C										
Drift rate μΩ	-0.02	-0.02	-0.01	0.02	0	0.03				
Drift rate mK	-0.02	-0.02	-0.01	0.02	0	0.03				
Residual standard deviation, mK	0.14	0.34	0.52	0.41	0.36	0.27				
Degrees of freedom	12	12	12	12	12	12				

It has been pointed out by Curtis [7] that drift rates for very long-term (>1000 hours) experiments often differ substantially from the drift rates for shorter-term experiments. Time did not permit us to test these thermometers beyond a few hundred hours but it would seem unlikely that there would be much difference in the drift rates for long and short-term heat treatments because most of the thermometers seemed relatively stable. Grain growth could cause a change in the drift rate with time. However, several of the birdcage thermometers were old and three had been at 960 °C and above for at least 300 hours before these tests.

It would appear from the results that with careful monitoring and sufficient annealing, the long-term stability of the cross thermometers and of four birdcage thermometers is satisfactory, but the stabilities of thermometers HTSS-19 and HTSS-16 are not good. One can say little at this time about the tungsten resistance thermometer, except that better initial stabilization procedures must be found and that with proper stabilization, its performance could approach the birdcage thermometers.

3. Stability of Thermometer Resistance at the Freezing Point of Zinc

3.1. Equipment and Procedures

We have recently installed a new type of freezing-point-of-zinc cell. Because of earlier problems with contamination of cells [3], it was decided that hermetically sealed cells were necessary. The cell (fig. 9) was made from borosilicate glass and the crucible and thermometer well were made from high-purity graphite. In addition to being shorter in overall length and offering a greater depth of immersion than the old cell, the new cell has a double thermometer well. Next to the zinc is a graphite well, and between that well and the thermometer sheath is a borosilicate-glass well which is an integral part of the cell. We have not noticed any particular problems of inadequate immersion or heat transfer because of this double well arrangement. Melting curves indicate that the zinc in the new cell is not as pure as the zinc in the old cell (which is not hermetically sealed).

In addition to protecting the zinc from contamination, the new cell design has allowed us to modify our low temperature furnace. Because of the small size of the cell, the aluminum moderating block in the furnace could be removed and replaced with an aluminum "can" (fig. 9). The zinc cell was placed inside this can. In this way, the cell was surrounded on all sides by aluminum. The furnace windings were simplified and now only a single winding is used. The old furnace and zinc cell are described in [3]. Initial tests with a 25-Ω standard platinum resistance thermometer indicated that the thermometer can track the temperature gradient due to the hydrostatic pressure of the liquid metal over 7 cm to within 20 μK.

In the course of testing the new zinc cell, we also gathered some data on the performance of the thermometers. Initial resistance measurements at the zinc point were taken with the birdcage thermometers after their stability tests at 960 °C, and the final measurements at the zinc point were taken after the tests at 900 °C. The resistances of the cross thermometers were studied at the zinc point before and after exposure at 960 °C. Occasionally, consecutive measurements were taken during one freeze. During this procedure, the thermometer was allowed to attain thermal equilibrium with the freezing zinc, resistance readings were taken, and the thermometer was removed for five minutes. The thermometer was

then reinserted and the process repeated. Up to three sets of readings were taken during a single freeze. Sometimes, several thermometers were tested during one freeze. The same procedure was followed with the exception that a different thermometer was introduced into the zinc-point cell after the five minute wait.

3.2. Results

The changes in thermometer resistances at the zinc point, in mK equivalents, are summarized in table 3. The birdcage thermometers had an initial shift in their zinc-point resistances of -1.2 mK to 9.9 mK. These values are based on an average of the observed changes in terms of R_{Zn}, W_{Zn}, and D_{Zn}, where R_{Zn} is the thermometer resistance at the freezing point of zinc,

$$W_{Zn} = R_{Zn}/R_0,$$

$$\text{and } D_{Zn} = R_{Zn} - R_0.$$

R_0 is the average of the ice-point resistances read before and after the zinc point. In some cases, the ice-point resistance changed more than the zinc-point resistance but no pattern emerged for the four birdcage thermometers. After the shift, the thermometers stabilized but they were not quite as stable as the cross thermometers.

Table 3. Zinc Point Data

Difference from preceding zinc point, $X_{i+1} - X_i$, mK

RT	ΔR_{Zn}	ΔW_{Zn}	ΔD_{Zn}	ΔR_0
HTSS-14	0*	0*	0*	0*
	-4.6	11.9	1.8	-5.7
	1.7	1.9	1.8	-0.1
	0 (a)	0	0	0
	-0.8	- 0.8	-0.8	0
HTSS-16	0*	0*	0*	0*
	4.4	16.3	9.0	-4.1
	0.4	6.4	2.7	-2.1
	-0.3	- 0.3	-0.2	0
	-0.4	- 0.4	-0.5	0
HTSS-21	0*	0*	0*	0*
	8.3	9.7	8.8	-0.4
	0.6	0.6	0.6	0
	0.7	0.7	0.7	0
	-0.3	- 0.3	-0.2	0
HTSS-22	0*	0*	0*	0*
	9.9	-14.2	0.5	-8.2
	0.3	0.4	0.4	0
	0.1	0.1	0	0
HTFQ-23	0**	0**	0**	0**
	1.0**	1.1**	1.0**	0**
	17.1	34.2	23.8	-5.8
	0.5	0.9	0.7	-0.1
	- 2.4	- 2.3	- 2.4	0
	3.0	2.6	2.9	0.2
	0.2	0.6	0.3	-0.1
	0.2	0.1	0.2	0
	0.3	0.3	0.3	0
	-0.2	- 0.2	-0.2	0

Table 3 (continued)

RT	ΔR_{Zn}	ΔW_{Zn}	ΔD_{Zn}	ΔR_0
HTFQ-24	0**	0**	0**	0**
	0.5**	0.1**	0.3**	0.1**
	26.9	30.0	28.1	-1.0
	- 3.0	- 2.9	- 2.9	0
	3.8	4.0	3.9	-0.1
	0.1	0.3	0.2	-0.1
	- 0.2	0.3	0	-0.2
	0.6	0.7	0.6	0
	- 0.2	- 0.5	-0.3	0.1
	0.2	0.2	0.2	0
	0	0	0	0
HTFQ-25	0**	0**	0**	0**
	1.3**	1.1**	1.2**	0**
	17.9	32.0	23.4	-4.8
	0.2	0.4	0.3	-0.1
	- 0.5	- 0.2	- 0.4	0.1
	0.6	0.8	0.7	-0.1
	0.1	- 0.1	0	-0.1
	0.1	0.1	0.1	0.1
	0.1	0.1	0.1	0

* data taken before exposure to 900 °C

** data taken before exposure to 960 °C

(a) bracketed runs were taken during the same freeze.

The cross thermometers showed a large initial increase of 20 mK to 30 mK in the zinc-point resistance. Because the ice-point readings remained relatively steady over the entire time that zinc-point readings were taken, strain and improper annealing do not seem to be the source of all the instability. It is particularly noteworthy that the ice-point resistance decreased while the zinc-point resistance increased. This would definitely point away from strain as the source of the change. Part of the increase may be due to the increasing devitrification of the initially clear silica glass sheath which results in the reduction of the radiation "piped" up the sheath. McLaren [8] has shown that errors equal to or exceeding 16 mK may be due to radiation losses up the stem of a standard 25-Ω thermometer. However, we might expect to find less than this because the sheaths devitrified only slightly during testing at 960 °C.

Another source of the observed change might be a change in the pressure in the zinc cell. If a leak had developed, the cell pressure would tend to approach one atmosphere at room temperature and 2.3 atmospheres at 420 °C. However, a pressure change of 1.3 atmospheres would cause the zinc point to change by 5.6 mK and this does not explain our large shift.

Perhaps the zinc had somehow become contaminated between the last measurements with the birdcage thermometers and the last measurements with the cross thermometers. It was not removed from the furnace during the course of these measurements but let us postulate that something unusual happened. A 25-Ω thermometer was used to monitor the zinc point over the eight month period in question, and it showed a total change in the zinc point of 0.7 mK. Therefore, it seems clear that the thermometers have changed. We do not know whether they changed gradually during the 300 hours at 960 °C or suddenly. In either case, we conclude that it may be necessary to monitor the zinc point or some other suitable fixed point, as

well as the triple point of water, during long-term stability tests.

After the shift in the resistance at the zinc point had occurred, the thermometers behaved in a stable manner. The fifth zinc point on HTFQ-23 and the fourth on HTFQ-24 (table 3) were measured consecutively during a continuous freeze. The plateau began to fall off shortly after HTFQ-24 was inserted. Previously, plateaux had usually lasted two hours but this one began to decrease after only one hour. All the following zinc-point measurements agreed with the first zinc points after the initial shift, and it was felt that the freeze had not progressed properly. These two points met the Dixon criterion [9] for rejection for α = 0.01 and so the runs were discounted.

Values of the zinc-point resistance of the cross thermometers following the large initial shift (with the exception of the two measurements noted above) were quite well behaved. The maximum observed change within this stable subset of readings was 1.1 mK and most of the changes were considerably smaller and positive. This is in keeping with our observations of the birdcage thermometers. The average changes in the resistance at the zinc point (based on an average of R_{Zn}, W_{Zn}, and D_{Zn}) from one freeze to the next were 0.02, 0.2 and 0.1 mK for the three cross thermometers with standard deviations of 0.3, 0.4, and 0.5 mK, respectively.

We do not know whether the changes in the zinc-point resistances of the cross thermometers following the work at 960 °C were larger than the changes in the zinc-point resistances of the birdcage thermometers because the intermediate temperature was 960 °C instead of 900 °C in the case of the birdcage thermometers or because there is some inherent characteristic in the cross thermometers which caused the change. One way to check this would be to perform the experiment using 900 °C as the intermediate temperature for the cross thermometers or using 960 °C for the birdcage thermometers.

4. Investigations of Thermometer Insulation Resistance

The birdcage thermometer was designed to provide a strain-free element and a small resistance which would reduce the effects of the electrical insulation leakage that shunts the sensor. A 25-Ω thermometer must have insulation resistance in the lead assembly and coil support of at least 25 MΩ at the triple point of water if the effect of the shunting is to be less than 1 in 10^6. A 0.25-Ω thermometer needs only 1 MΩ of insulation resistance at the gold point to keep the effects of shunting below 1 in 10^6.

4.1. Equipment and Procedures

To check the insulation resistance of the cross thermometers, a special thermometer was constructed. Its construction was similar to the other cross thermometers, except that the thermometer element was cut at its mid-point. In this way we could measure, under realistic conditions, the insulation resistance of the element support and lead assembly as a function of temperature, frequency, and voltage. A less extensive investigation was carried out with a birdcage thermometer (HTSS-22), which had a fifth "lead" wire running from just above (but not touching) the sensing element to outside the thermometer head. The measurements, though not as ideal as measurements with the cross thermometer, were useful.

Two measurement systems were used. For the higher frequencies (50 to 1000 hz), a capacitance bridge was used and resistance values were read from the bridge. For the lower frequencies (10^{-3} to 10^{-1} hz), the circuit shown in figure 10 was used. R_T is the shunting insulation resistance, and e is the measured voltage across a 100 Ω resistor. The leakage current through the thermometer is supplied by a dry cell and is i_L = e/100. Hence, the leakage resistance, R_T, is given by

$$R_T = V/i_L = 100V/e.$$

Measurements of e were made by connecting a strip chart recorder across the 100 Ω resistor. The voltage V, applied across the thermometer, was established using a resistor of known value (10^5 or 10^6 Ω) in place of R_T and adjusting the slidewire. The current was reversed by manual switching. All measurements at 600 °C and above were made in the gold-point cell

to approximate the conditions of actual use. The resistances at 0 °C were determined from measurements taken in a triple-point-of-water cell.

4.2. Results

Figures 11 and 12 give the results of these tests for the cross thermometer. It would seem that an insulation resistance of 1 MΩ is not as easily achieved as we had initially thought [3]. Below 850 °C, the insulation resistance exceeded 1 MΩ. There was very little dependence on frequency or voltage until the temperature dropped below 700 °C. Thus, the frequency dependence of the insulation resistance is a problem with ac resistance bridges. These results indicate that researchers should make measurements whenever possible to determine the magnitude of shunting errors under their operating procedures.

Figure 12 presents typical sets of data on insulation resistance for two temperatures at several frequencies and voltages. For the lower temperatures, there is a larger difference between the 10 V and 1 V readings than is observed at the higher temperatures. Although higher voltages yielded higher insulation resistances, voltages above 1 V should not be used. Since the insulators do not obey Ohm's law, voltages should be kept as small as is practical. Although the actual value used in thermometry is less than 1 V, we see that the change between 1 V and 0.1 V is small so the value chosen below 1 V is not critical.

The insulation resistances measured in the birdcage thermometer were of the same order of magnitude as the cross thermometer resistances. The resistance at 1065 °C was 0.5 MΩ at frequencies between 50 and 1000 hz. The insulation resistance at 0 °C is given in table 4. There is less than an order of magnitude difference between the two types of thermometers.

Table 4. Insulation Resistance of HTSS-22 at 0 °C

Frequency, hz	Resistance, MΩ
50	8.4×10^3
100	2.1×10^4
200	1.0×10^4
400	4.7×10^3
1000	1.8×10^3

We observed considerable polarization of the insulators at the low frequencies; the initial resistance after the switch was reversed changed exponentially with time. The effects of polarization were not examined at higher frequencies ($\geqq$ 50 hz) but some polarization may have occurred.

Additional work is planned with different configurations and materials for thermometer elements. The frequency dependence of the insulation resistance is expected to be a problem. Therefore, more data on other insulators, e.g., BeO, are needed before ac resistance bridges can become standard laboratory instruments. More data must be taken in the mid-frequency range (0.01 to 50 hz) because the frequency-resistance curve appears to break in this region.

5. Summary

The results of the stability tests were, for the most part, encouraging. All drift rates of three types of thermometer as a result of heat treatments at the temperatures discussed were equal to or less than 0.3 mK/h and most were less than 0.1 mK/h. The residual

standard deviations of most of the thermometers about the straight line fit to the data were small. It appears that the cross thermometers were slightly more stable and less subject to drift than the birdcage thermometers. The stability of thermometer HTSS-15 was outstanding among the birdcage thermometers (even among the cross thermometers). The results of the heat treatments of thermometers HTSS-16 at 1065 °C and HTSS-19 at 960 °C showed them to be the least stable and the least satisfactory.

The reproducibility of the cross thermometers at the high temperature fixed points (silver and gold) will have to be tested. They have not been cycled "rapidly" (>20 hours of exposure to high temperatures) nor have they been quenched before annealing with the exception of two or three sets of measurements at the silver point taken with each thermometer after the second set of measurements at the zinc point. The results of these measurements are given in table 5.

Table 5. **Silver** Point Data

Difference from preceding silver point, $X_{i+1} - X_i$, mK

RT S/N	ΔR_{Ag}	ΔW_{Ag}	ΔD_{Ag}	ΔR_0
HTFQ-23	0 (a)	0	0	0
	1.9 (a)	1.8	1.9	0
	-0.8	-1.5	-1.0	0.1
	2.1	2.3	2.1	0
HTFQ-24	0	0	0	0
	0.6	0.4	0.6	0
HTFQ-25	0	0	0	0
	-0.5	-0.4	-0.3	-0.1

(a) bracketed runs were taken during the same freeze.

The birdcage thermometers were not as steady at the zinc point as the cross thermometers but they did not exhibit the large initial shift found with the cross thermometers. Further research will have to be carried out to probe the mechanism of the shift. Thermometer resistances at the zinc point (and perhaps the tin point) should be monitored more frequently during exploratory stability tests at 960 °C and above.

Insulation leakage is a problem. The frequency dependence of the leakage must be carefully investigated in light of the increasing popularity of ac resistance bridges. Work is planned to investigate both silica glass and sapphire in both the birdcage and cross configurations. Efforts will be made to determine the effect of the lead assembly configuration on frequency dependence. The drop in insulation resistance below 1 MΩ at high temperatures is disturbing. New insulators should be tested. An effort should be made to determine how much leakage is through the leads and how much is through the element support.

At this stage, we can really say little about the suitability of our tungsten thermometer. It seems to be approaching the stability of the birdcage thermometers and could surpass their stability. Certainly, further work is merited on the investigation of initial stabilization and coil configurations.

It is clear from our results that several projects should be continued or started. Stability testing should continue with more frequent monitoring of the thermometers at low temperature fixed points. New insulator and element configuration combinations should be tested for stability and insulation resistance. Work should continue with tungsten and new materials for thermometer elements should be investigated.

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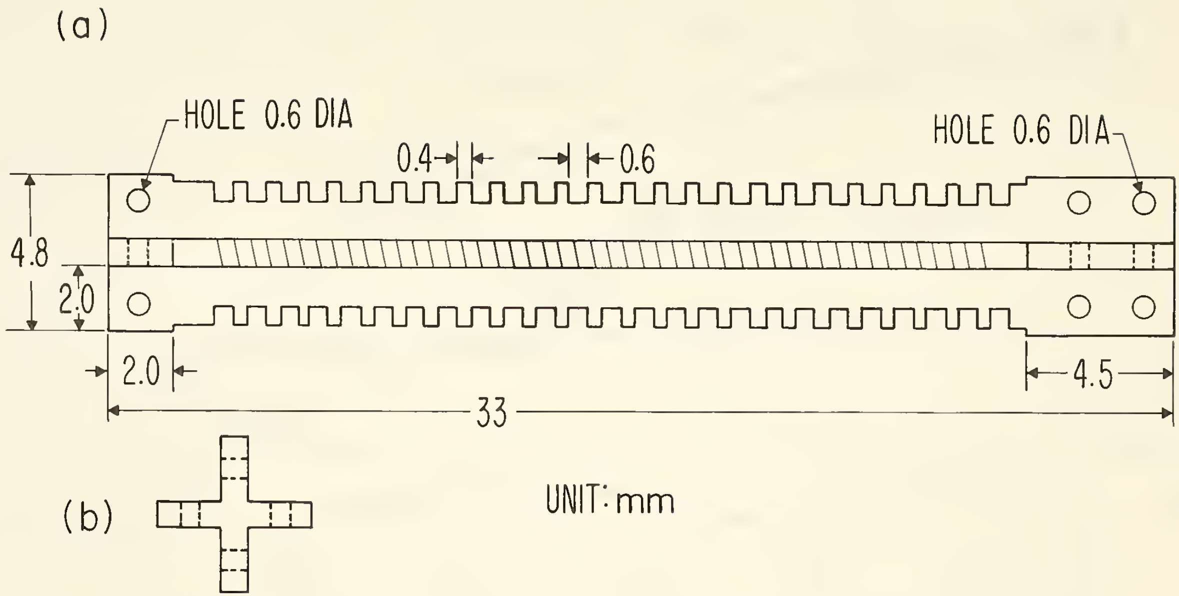

Figure 1. Details of the silica-glass support for the cross (HTFQ) thermometers. (a) side view. (b) end view.

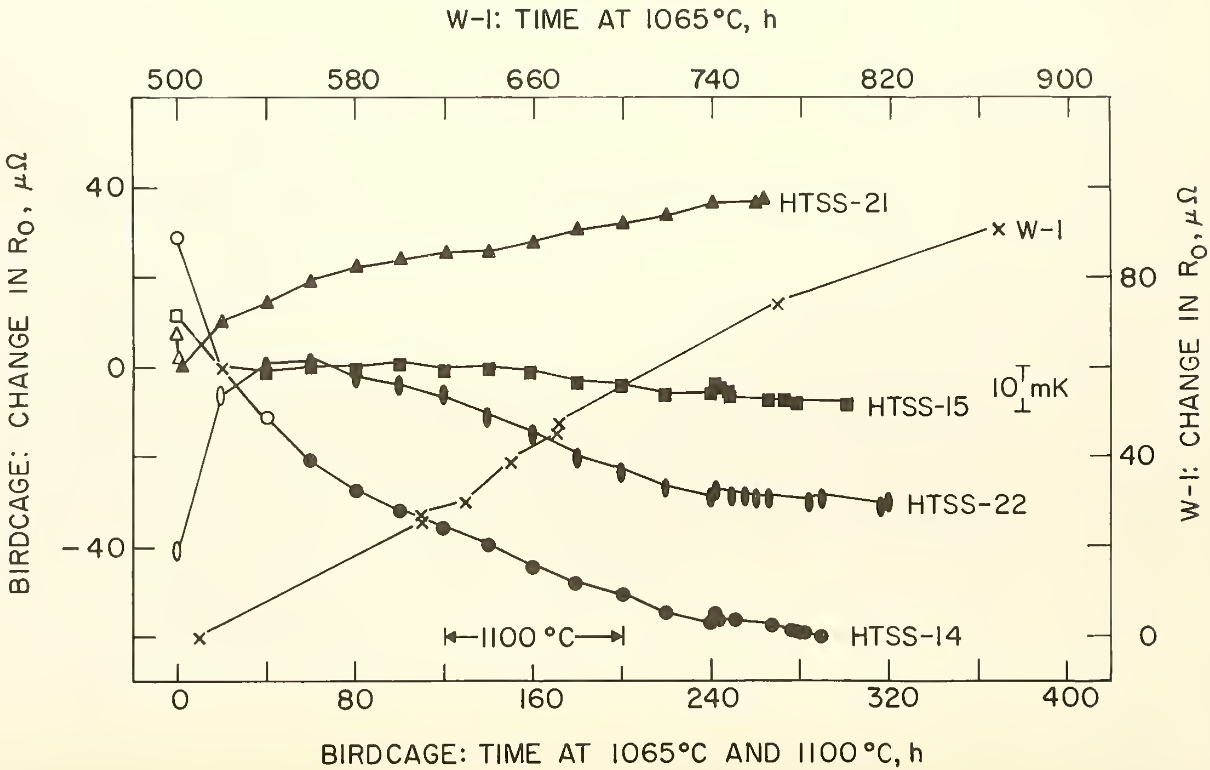

Figure 2. Behavior of four birdcage (HTSS) thermometers and thermometer W-1 with exposure to temperatures in the region of 1065 °C. Open symbols were not used in computing drift rates. The temperature scale does not apply strictly to W-1.

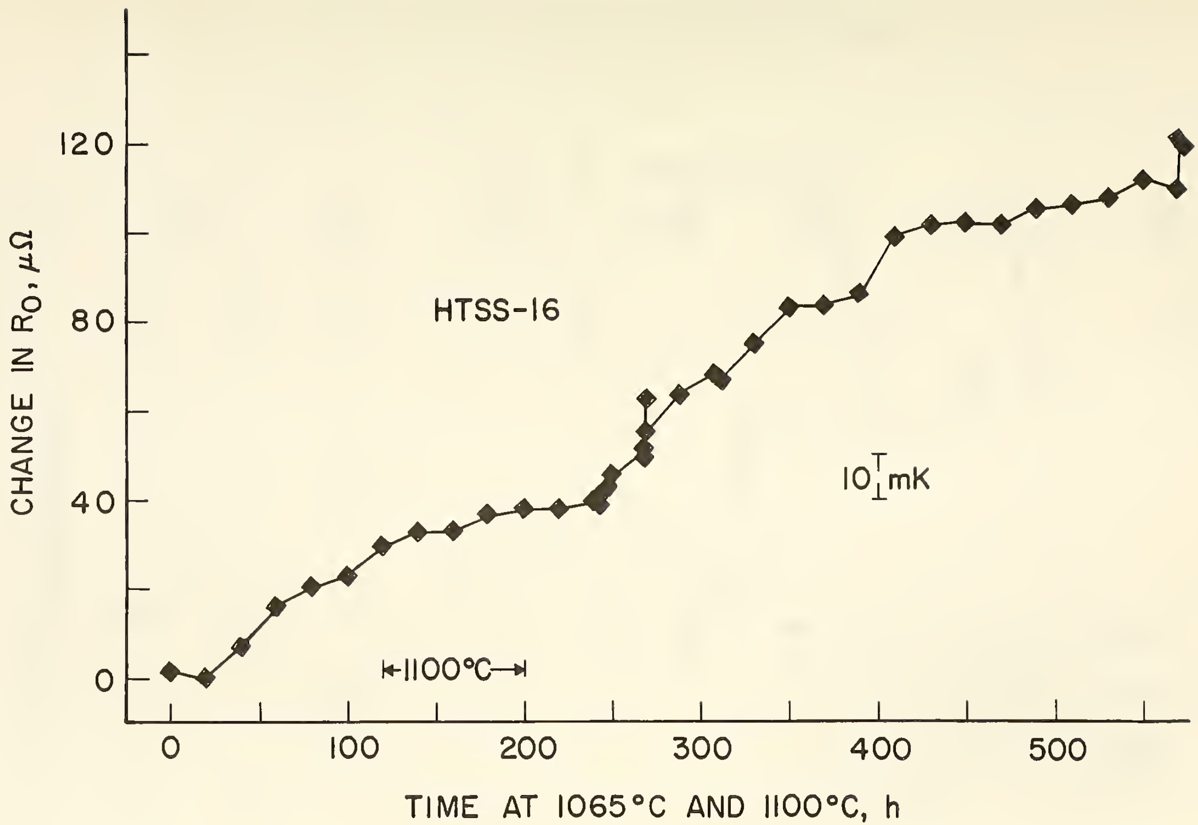

Figure 3. Behavior of birdcage thermometer HTSS-16 with exposure to temperatures in the region of 1065 °C. The first point was not used in computing the drift rate.

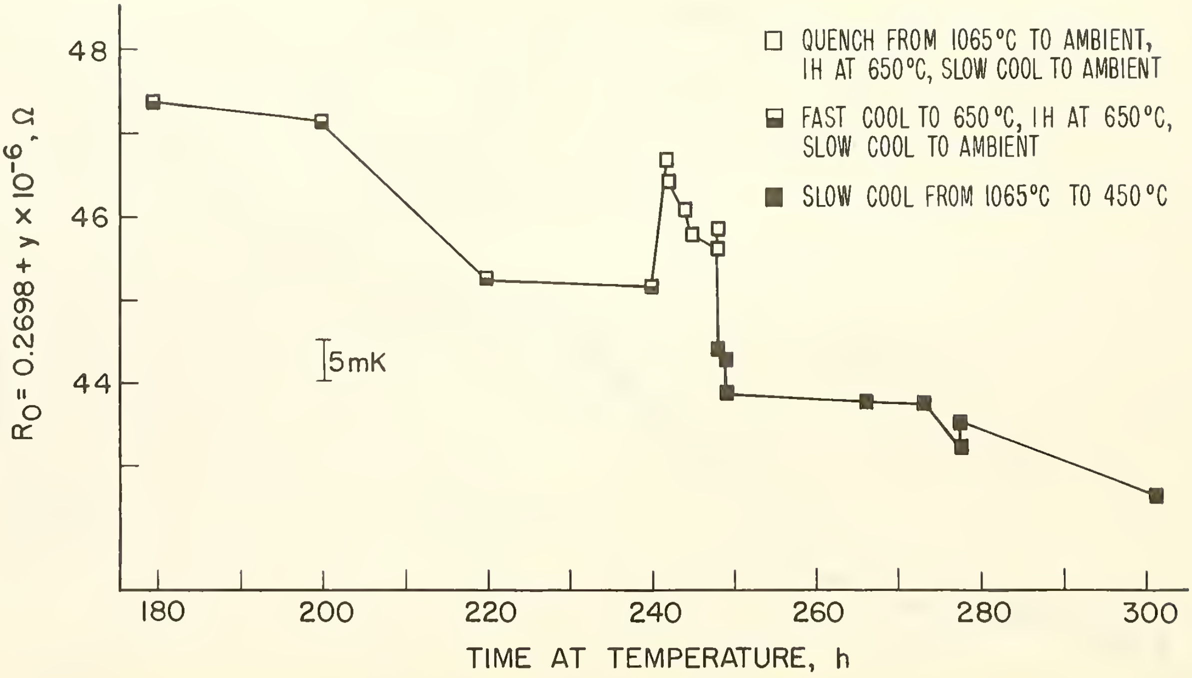

Figure 4. Effects of various annealing patterns on birdcage thermometer HTSS-15 after heating in the region of 1065 °C.

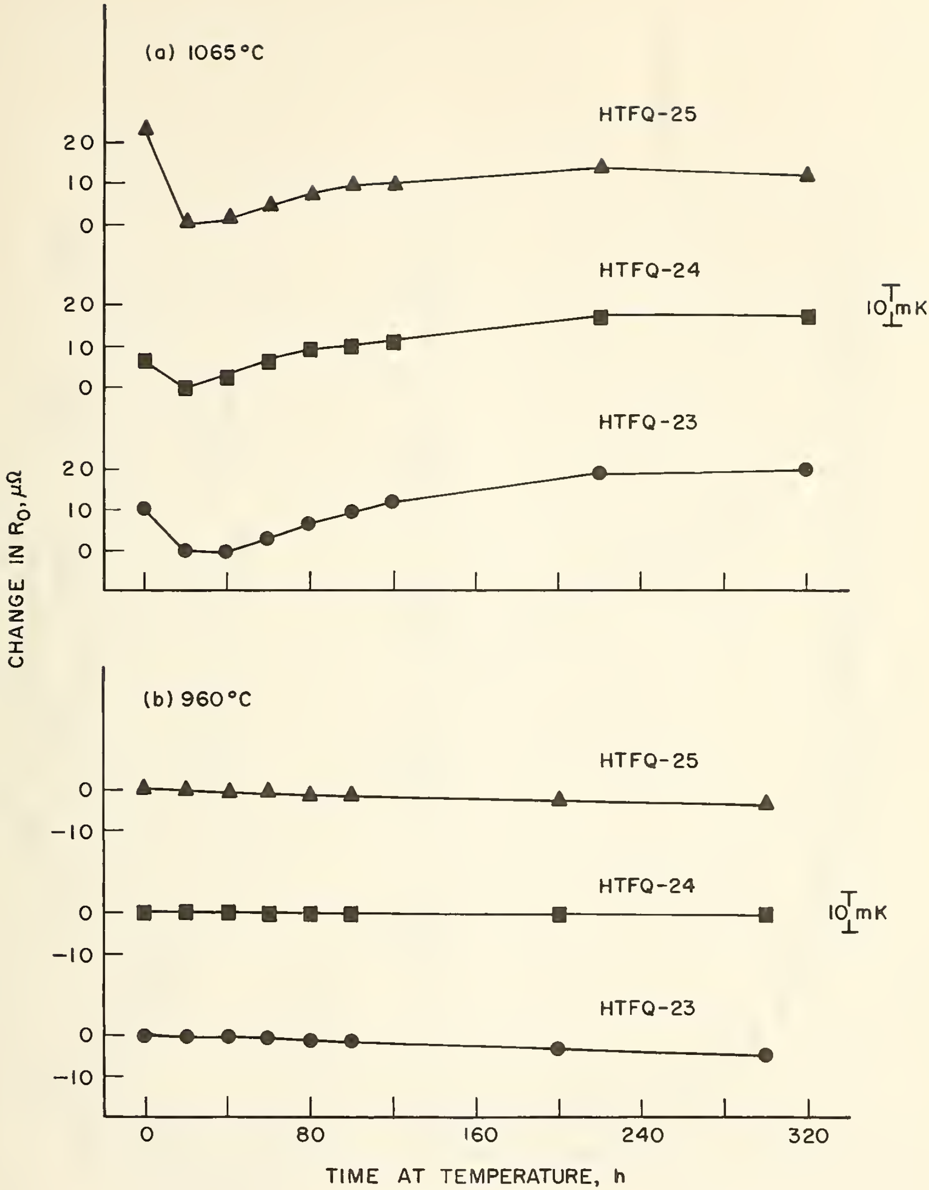

Figure 5. Behavior of cross (HTFQ) thermometers. (a) Exposed to 1065 °C. The first point in each case was not used in computing the drift rate. (b) Exposed to 960 °C.

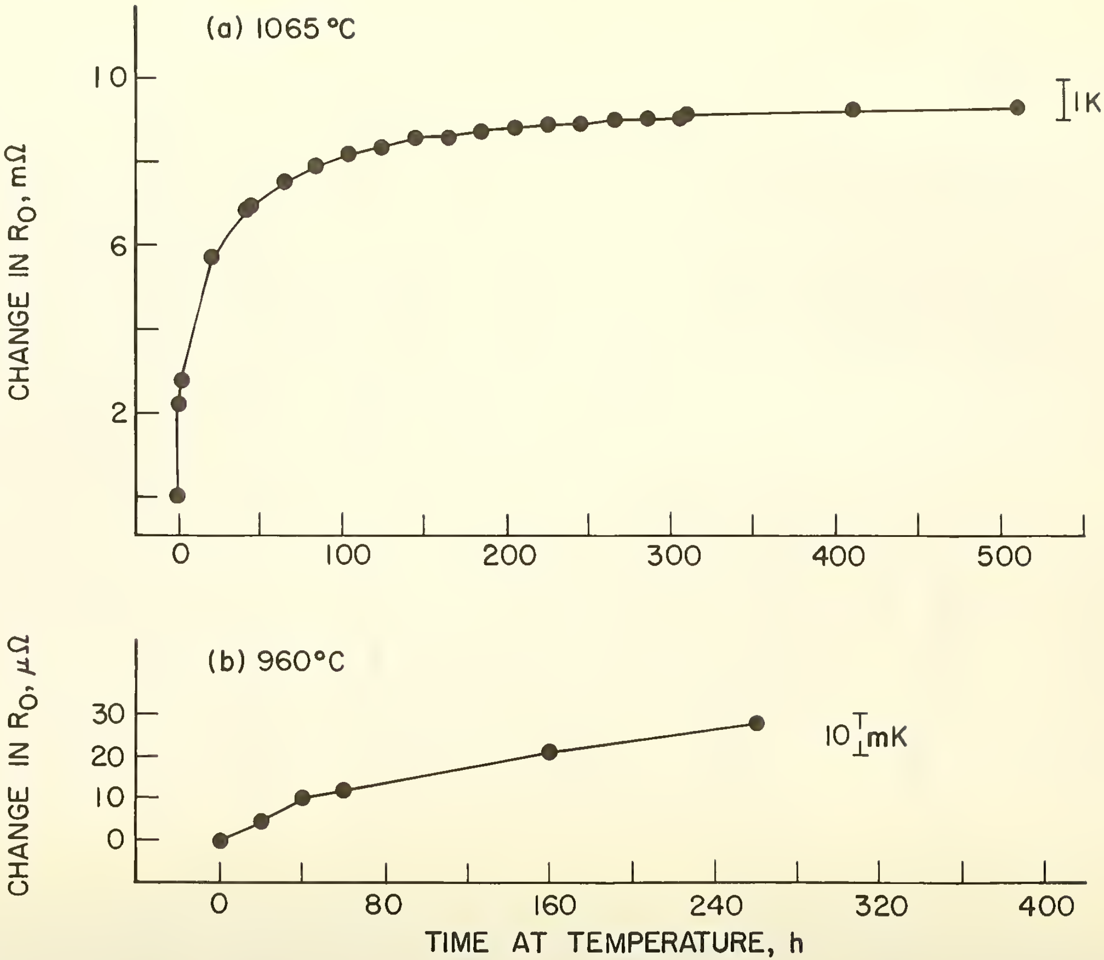

Figure 6. Behavior of the tungsten thermometer W-1. (a) Exposed at 1065 °C. (b) Exposed at 960 °C. Note different scales.

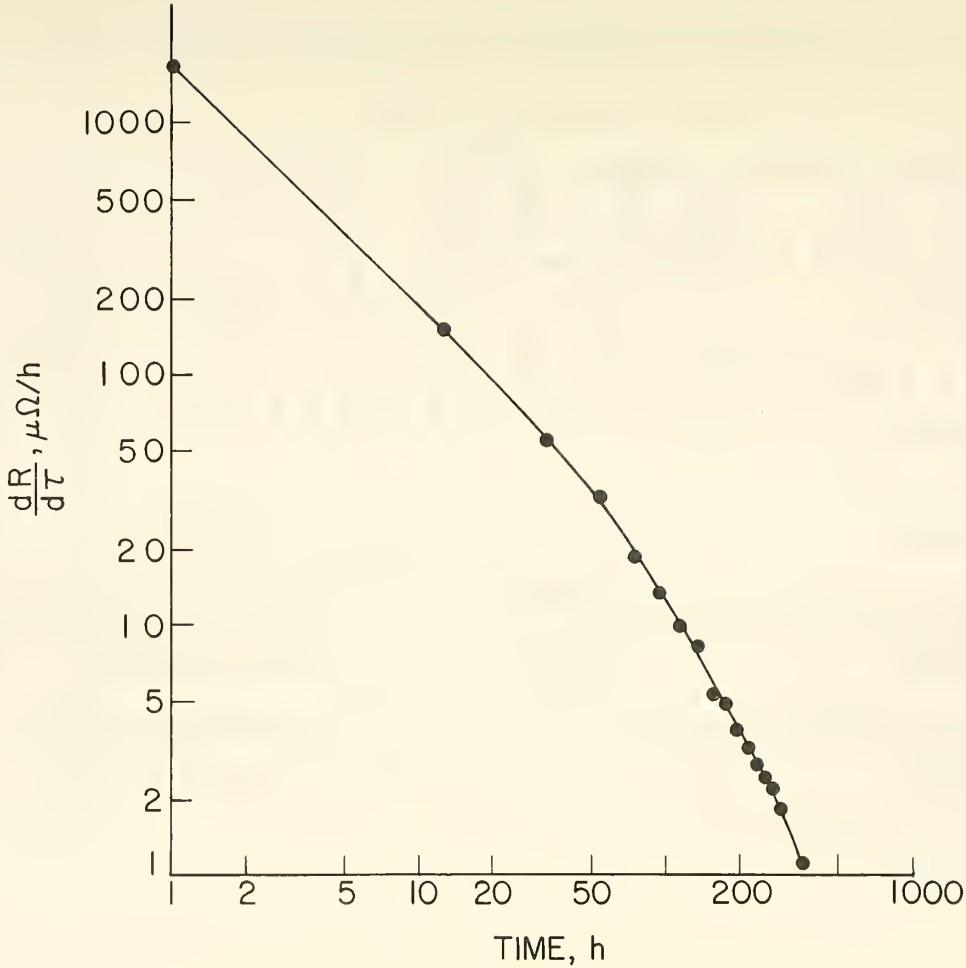

Figure 7. Drift rate of W-1 as a function of time spent at 1065 °C.

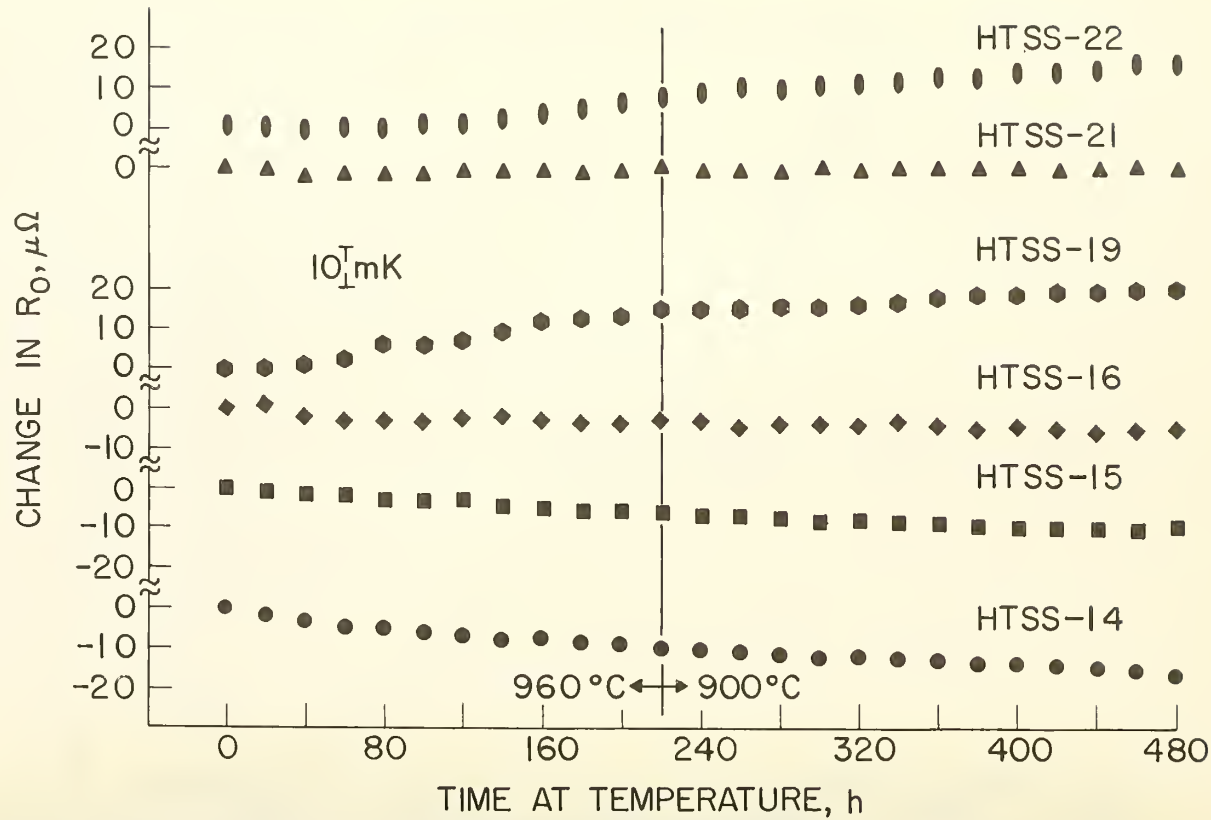

Figure 8. Behavior of birdcage thermometers (HTSS) after exposure at 960 °C and 900 °C.

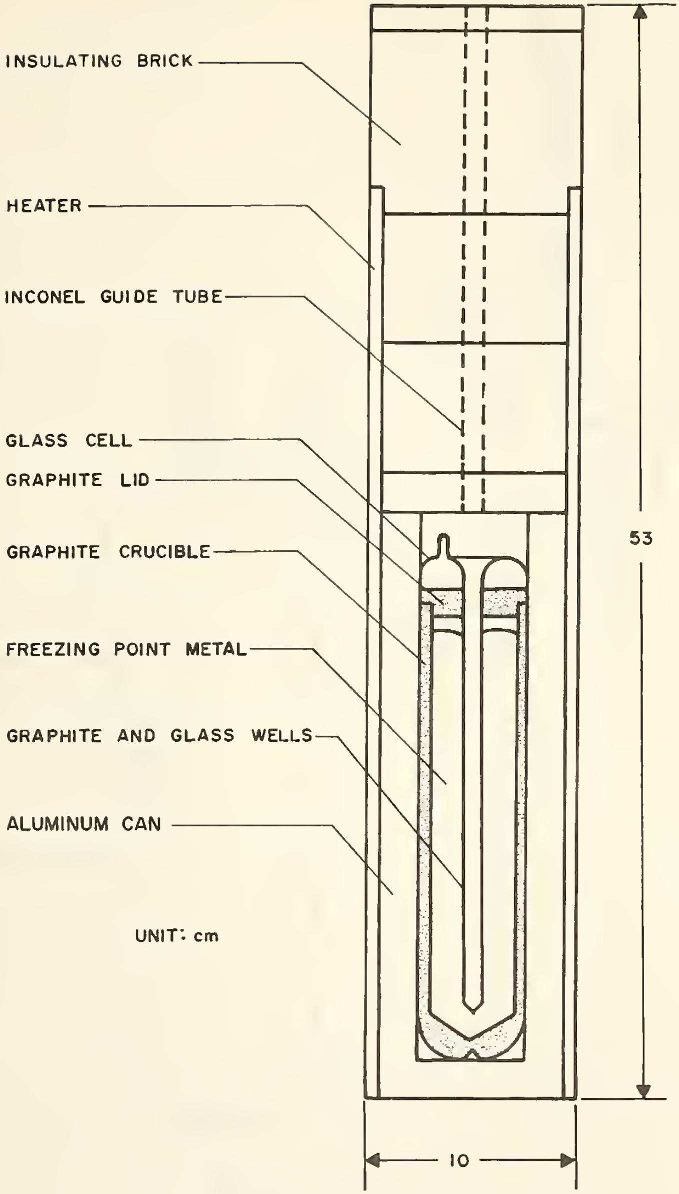

Figure 9. Details of the construction of the core of the low temperature furnace and of the hermetically sealed freezing-point-of-zinc cell.

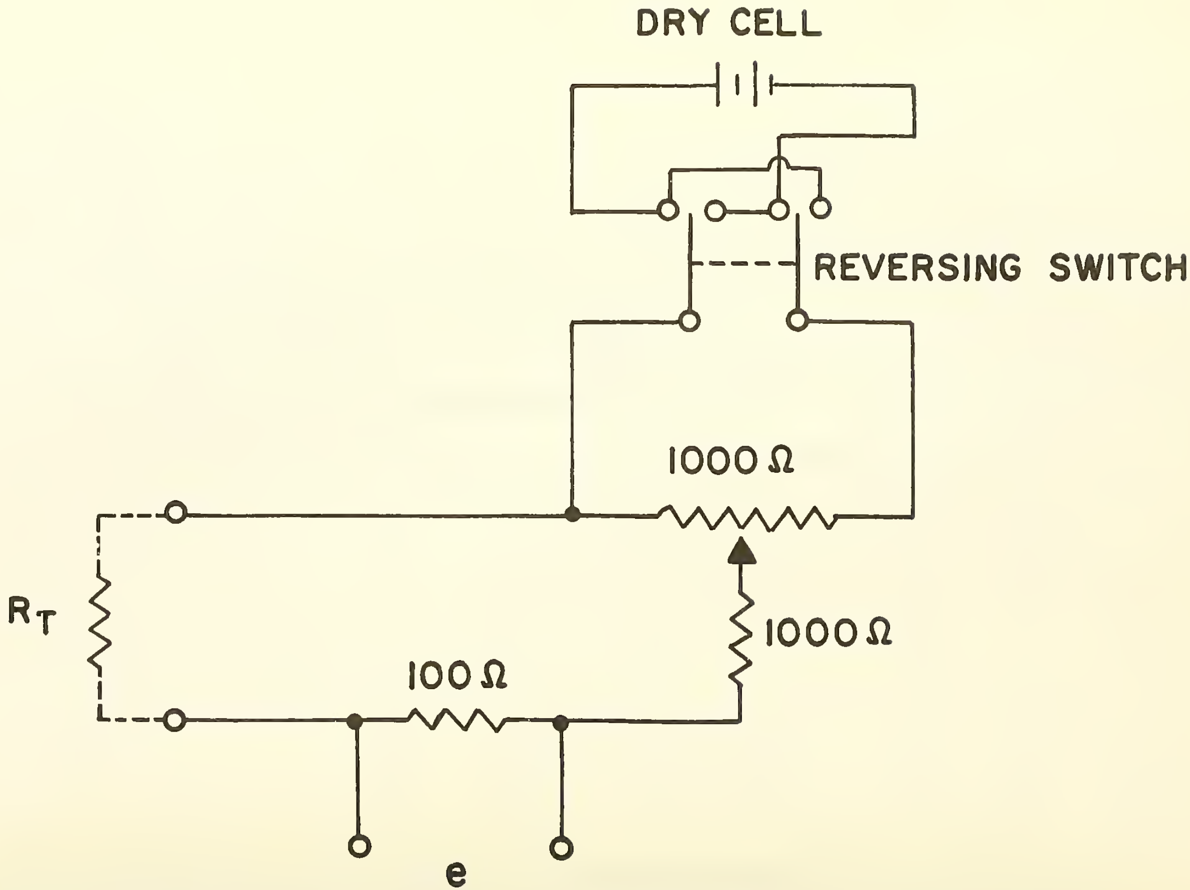

Figure 10. Circuit diagram of the apparatus used to measure insulation resistance at low frequencies. R_T is the leakage resistance being measured, e is the point at which the leakage current is measured.

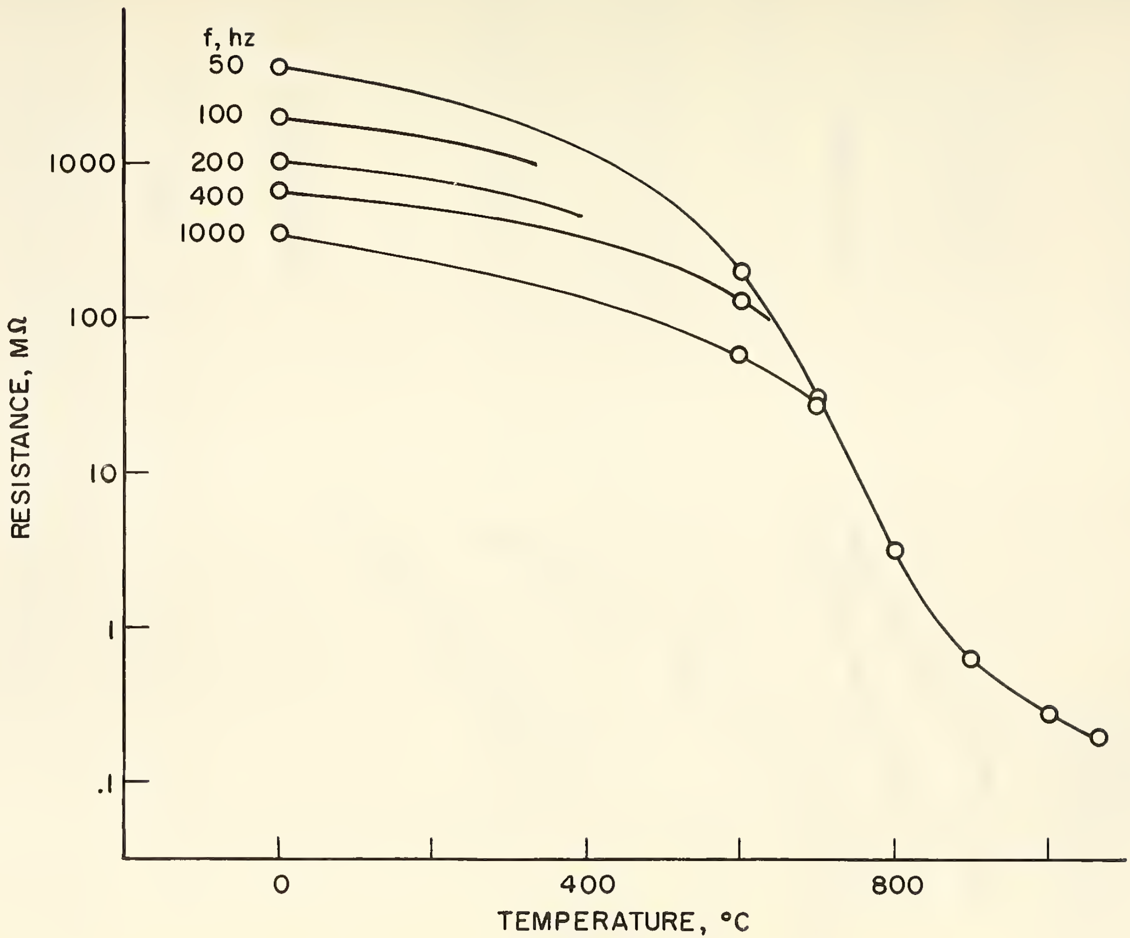

Figure 11. Summary of tests of insulation resistance of HTFQ-23.

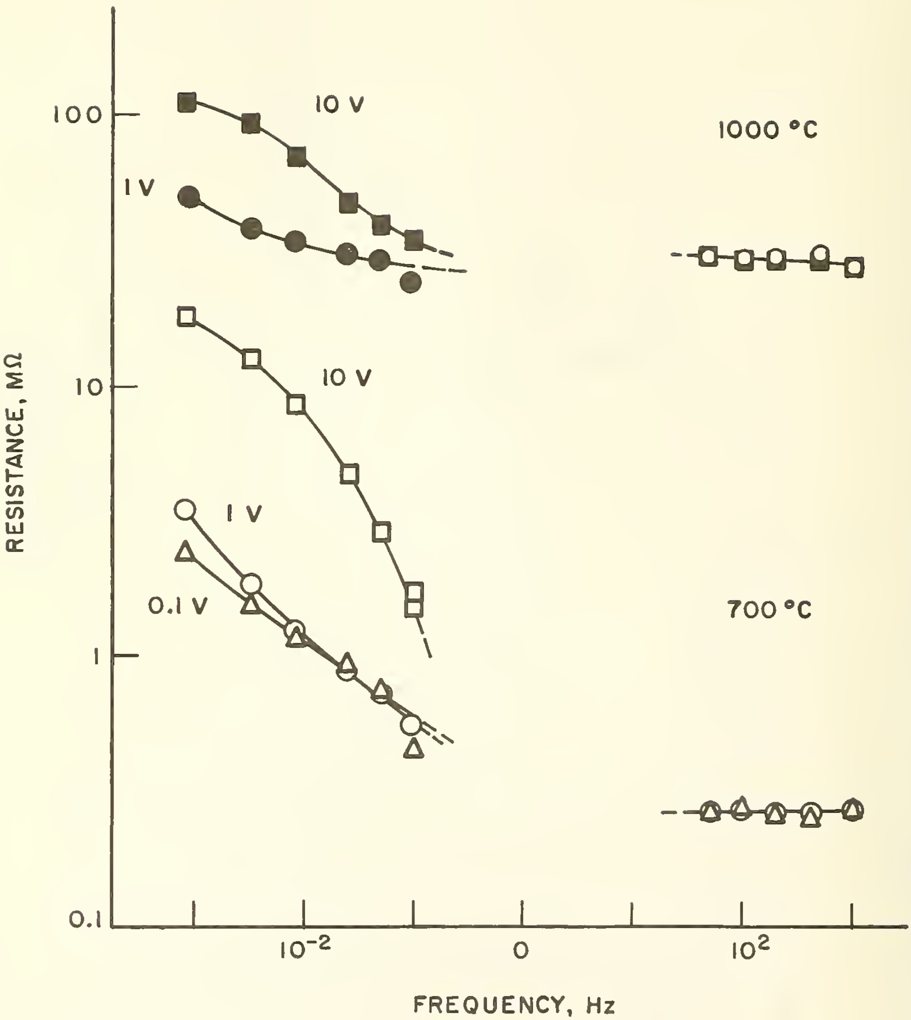

Figure 12. Typical sets of data for the insulation resistance of HTFQ-23 at two temperatures and several voltages.

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Data are presented concerning the stability of high temperature resistance thermometers. Two types of platinum and one type of tungsten thermometer were tested at 1065 °C and 960 °C. Some of the platinum thermometers were also tested at 900 °C and at the freezing point of zinc. Results of tests are also given on the insulation resistance of sensor supports and lead assemblies for the two types of platinum thermometers. A new design for a zinc-point cell is discussed briefly. Suggestions for future work are indicated.

17. KEY WORDS (Alphabetical order, separated by semicolons) Freezing point of zinc; high temperature resistance thermometer; insulation resistance; platinum resistance thermometer; resistance thermometer; stability; tungsten thermometer; zinc.

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